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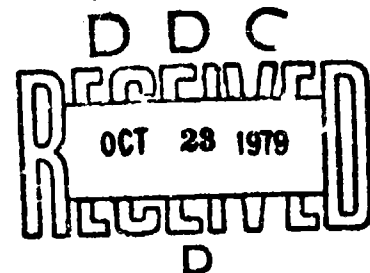
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TECHNICAL REPORT ARLCD-TR-78062

**DETONATION OF GUANIDINE NITRATE  
AND NITROGUANIDINE  
MANUFACTURED VIA U/AN AND BAF PROCESSES**

J. WENDELL LEACH

AUGUST 1979



**US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND  
LARGE CALIBER  
WEAPON SYSTEMS LABORATORY  
DOVER, NEW JERSEY**

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\* The attached page 34 (fig. 9) should be substituted for the original page appearing in the technical report identified above.

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19. KEY WORDS (Continue on reverse side if necessary and identify by block number) <table border="0"> <tr> <td>U/AN - Urea/Ammonium Nitrate</td> <td>Propagation</td> <td>Welland Process</td> </tr> <tr> <td>BAF- British Aqueous Fusion</td> <td>Sensitivity</td> <td>Guanidine nitrate</td> </tr> <tr> <td>Critical diameter</td> <td>Detonation</td> <td>Hazard analysis</td> </tr> <tr> <td>Thermal analysis</td> <td>Nitroguanidine</td> <td>Propellant</td> </tr> <tr> <td></td> <td></td> <td>Explosive</td> </tr> </table>			U/AN - Urea/Ammonium Nitrate	Propagation	Welland Process	BAF- British Aqueous Fusion	Sensitivity	Guanidine nitrate	Critical diameter	Detonation	Hazard analysis	Thermal analysis	Nitroguanidine	Propellant			Explosive
U/AN - Urea/Ammonium Nitrate	Propagation	Welland Process															
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Thermal analysis	Nitroguanidine	Propellant															
		Explosive															
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) <p>The objective was to derive detonation data for hazards analysis and for the related safety design of facilities for manufacturing nitroguanidine by the urea/ammonium nitrate (U/AN) and the British aqueous fusion (BAF) processes. Critical diameter, propagation, sensitivity, and thermal characteristics of a number of mixtures and compounds, representative of selected streams in the processes, were determined.</p>																	

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20. Abstract (continued)

Critical diameter tests indicate that streams from the Evaporator Outlet, Mixed Reactor Feed, and the Liquid Reactor Outlet of the U/AN process will propagate when initiated with a booster and that they are mass-detonable. Thermal analysis tests on the stream mixtures indicate that they do not react violently when being heated to elevated temperatures but they do thermally decompose under these conditions.

Propagation test results show that certain streams, peculiar to the U/AN process, propagate when detonated. However, propagation in 5.08 cm (2 in.) pipes was not complete on any mixture containing 25 percent or more water. The results also show that the process streams in the wet guanidine nitrate buildings, used in the BAF process, are not detonable; this is also true of cold melts (molten mixtures allowed to cool) in event of plant shutdown.

The sensitivity (hazard) data on guanidine nitrate shows it is a relatively low-order explosive when compared to TNT, but that it is mass detonable.

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The author wishes to express his appreciation to the following persons and organizations for their extensive contributions to the detonation study of guanidine nitrate and nitroguanidine.

1. Mr. Thomas Caggiano and Mr. George Karshina, Office of the USAMC Project Manager for Production Base Modernization and Expansion.

2. Mr. C. H. Nichols, Office of Process Design Technology Branch, Manufacturing Technology Division, ARRADCOM, Dover, NJ.

In addition to providing technical assistance to the Hercules Powder Company in the design and operation of the pilot plant, they also conducted many tests on selected process streams associated with the U/AN (urea/ammonium nitrate) process and the BAF (British Aqueous Fusion) process for guanidine nitrate manufacture and subsequent nitroguanidine production.

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## INTRODUCTION

Improved procedures to produce nitroguanidine by the Urea/Ammonium Nitrate (U/AN) (fig. 1) and the British Aqueous Fusion (BAF) (fig. 2) processes were investigated under MM&T project 5714169 and were summarized in Technical Report 4566 (ref. 1). The current report presents detailed propagation, sensitivity, and dynamic explosive properties of various in-process streams related to the U/AN and BAF processes, in addition to guanidine nitrate and nitroguanidine. This data covers safety considerations related to the hazard analysis of the basic manufacturing processes and also of the BAF based production facility currently being erected at the Sunflower AAP under AMC project 5752632. The basic categories presented in the report cover the areas of critical diameter determination, differential thermal analysis (DTA) and thermal gravimetric analysis (TGA), and propagation and sensitivity determinations.

## PICATINNY ARSENAL TEST STUDIES

A series of tests were performed under MM&T project 5714169 by Picatinny Arsenal in an effort to establish criteria necessary for the manufacture and production of nitroguanidine.

### Critical Diameters

An important phase in the investigation of the U/AN process, figure 3, (ref. 6) was to determine the critical diameters for certain process streams which were suspected of being mass detonable. This data was required to develop design criteria for self-quenching detonation arrestors to reduce the potential loss of personnel and facilities, and to meet safety requirements. The design of an arrestor is normally based upon either an active method which uses a detonation trap, or a passive method based upon critical diameters. The passive method, which is the preferred method if realizable under MM&T project 5714169, is the approach used in this study. Critical diameter is defined as the largest diameter of pipe containing the explosive which shows no evidence of propagating an explosive reaction through the test specimen.

Critical diameter determinations were made for four mixtures simulating certain key streams in the U/AN process. The mixtures tested were as follows:

Composition coded #1 U/AN process, evaporator outlet, temperature 130°C:

Guanidine nitrate	13% by wt
Ammonium nitrate	74% by wt
Urea	13% by wt

Composition coded #2 U/AN process, mixed reactor feed, temperature 120°C:

Guanidine nitrate	9% by wt
Ammonium nitrate	60% by wt
Urea	31% by wt

Composition coded #3, U/AN process. liquid reactor outlet, temperature 180°C:

Guanidine nitrate	33% by wt
Ammonium nitrate	57% by wt
Urea	10% by wt

Composition coded #4, nitroguanidine processes, nitroguanidine reactor outlet, temperature 40°C:

Sulfuric acid	56% by wt
Water	7% by wt
Ammonium sulfate	14% by wt
Nitroguanidine	21% by wt
Guanidine nitrate	2% by wt

In testing for critical diameters, assemblies of two or more lengths of different diameter pipes (containing a test composition), joined end-to-end by reducing couplings were used. The length of each section of pipe was selected to assure that propagation would stop within the pipe. Each assembly was initiated with a combination of a C-4 booster in a 3/1: length/diameter ratio and a J-8 blasting cap. In assemblies up to 7.62 cm (3 in.) in diameter, a cylindrical booster 7.62 cm long and 2.54 cm in diameter was used. Assemblies of 10.16 cm (4 in.) in diameter were initiated with a 30.48 cm long by 10.16 cm in diameter conical booster. During some of the initial tests, a rack (fig. 4) was used which contained five pipe assemblies with C-4 boosters and one assembly without a booster.

The assembly without a booster was included to determine if sympathetic detonation would occur. After several initial tests with composition #1, the test rack was discarded because of the difficulty in maintaining and controlling the required test temperature. The rack was cumbersome and required too much time to set up and pour the material resulting in a heat loss. This necessitated reheating in some instances. The results of these tests are shown in table 1. No sympathetic detonation was shown in the firings.

The compositions were conditioned and detonated within  $\pm 5^{\circ}\text{C}$  of the specified temperature.

The critical diameter was established in accordance with the procedure specified in the CPIA publication No. 194, procedure 2.18 (ref. 4). Assemblies with progressively increasing diameters were initiated until a detonation was sustained and ceased at some diameter within the assembly. Assemblies with diameters greater and less than this diameter were evaluated until a total of three cessations of propagation at the same diameter were obtained. This diameter was established as the critical diameter. The results of these tests are shown in tables 1 and 2.

Fourteen tests were performed on composition coded #1 at  $130^{\circ}\text{C}$ . The mixture proved to be mass detonable and established a critical diameter of 2.54 cm (1 in.).

Composition coded #2 was tested at  $120^{\circ}\text{C}$  in six different pipe assemblies and a critical diameter of 3.81 cm ( $1\frac{1}{2}$  in.) was established. The third composition, #3, at a temperature of  $180^{\circ}\text{C}$  indicated a critical diameter of 2.54 cm (1 in.) after a total of eight assemblies were tested. Two assemblies consisting of a 50.8 cm (20 in.) long by 2.54 cm (1 in.) diameter pipe joined to a 30.48 cm (12 in.) long by 1.91 cm ( $\frac{3}{4}$  in.) diameter pipe were tested but did not propagate. Subsequently, two tests were made using assemblies with 45.72 cm (18 in.) long by 3.81 cm (1.5 in.) diameter pipe joined to a 30.48 cm (12 in.) long by 2.54 cm (1 in.) diameter pipe. Propagation occurred through the 3.81 cm (1.5 in.) diameter pipe but not through the 2.54 cm (1 in.) diameter pipe.

The composition coded #4, at  $40^{\circ}\text{C}$ , required more extensive testing to determine if a critical diameter existed for this mixture. The testing started with a 30.48 cm (12 in.) long by 3.81 cm (1.5 in.) diameter pipe and proceeded to a 91.44 cm (37 in.) long by 10.16 cm (4 in.) diameter pipe. In each instance pipe damage was evident but it was assumed to be the result of the C-4 booster and not the composition. To verify this assumption, a test was performed with a 66.04 cm (26 in.) long by 10.16 cm (4 in.) diameter pipe filled with water. The resultant damage was the same as the damage to the pipes tested previously using the mixture; consequently, it was concluded that this mixture is not mass detonable.

## Hazard Testing

A group of samples consisting of aqueous slurries of urea ammonium nitrate and guanidine nitrate were selected for friction and impact testing (ref. 7). Table 3 shows that five of the six samples did not initiate upon impact as per procedure outlined in Picatinny Arsenal Technical Report 3278, page 2. The sixth sample initiated on impact at a drop height of 38 inches which is probably due to the high ammonium nitrate content of the sample.

These samples were also Friction-Pendulum tested in accordance with instructions stated in Picatinny Arsenal Testing Manual #7-1. They showed no reaction with the steel shoe.

## Thermal Analysis

The mixtures described in the critical diameter tests section of this report (table 2) were also subjected to differential thermal analysis (DTA) and thermogravimetric analysis (TGA). The objective of these tests was to determine if reactants and reactant product mixtures, present in the various stages in the production of nitroguanidine, are capable of exploding during a heating cycle. Measurements were made with a DuPont 900 analyzer. Figures 5 through 8 contain the thermal data obtained.

The DTA method involves heating the material being analyzed, simultaneously with a thermally inert reference material, to elevated temperatures at a constant rate. The temperature difference between the test sample and the reference is continuously plotted versus temperature. The resultant exothermic and endothermic curves reveal unique characteristics of the material and its physical, chemical, and thermal reactions. The DTA is a continuous record of the thermal effects accompanying melting, boiling, crystalline transition, dehydration, decomposition, oxidation, and reduction and provides a qualitative study of the material.

The TGA method involves continuous weighing of the material under investigation as it is being heated at a constant rate. The weight loss of the test sample is continuously plotted versus temperature. The TGA procedure presents an excellent visual quantitative study of the observed changes.

None of the four mixtures detonated or burned while being subject to either test, as evidenced by the curves. The DTA analysis of the four compositions in the U/AN process is shown in table 4. In no instance during the determinations was there a violent reaction. The mixtures and components appeared to vaporize with some decomposition and reaction. However, the reactions appeared to be controlled.

### Propagation

Tests were performed to determine the explosive propagation characteristics of various streams according to procedure No. 2.18 in reference 4 that may be encountered in both the U/AN and BAF processes. Emphasis was placed upon evaluating the behavior of guanidine nitrate, guanidine nitrate/water, and nitroguanidine/water to determine the minimum water concentration that would sustain detonation. All tests, unless otherwise stated, were conducted in nominal 5.08 cm (2 in.) diameter pipes using a cylindrical C-4 booster with a length/diameter ratio of 3 to 1 and a J-8 blasting cap. Witness plates of 1.55 cm (3/8 in.) thick mild steel were used to assess propagative behavior. The detonation rate tests were conducted using the test setup shown in figure 9.

### U/AN Process Streams

The compositions and propagation results for the streams representing the guanidine nitrate (GN) crystallizer and the evaporator outlet are in table 5. The crystallizer stream did not propagate in 5.08 cm (2 in.) diameter pipe. The evaporator outlet stream gave complete detonation of 5.08 cm diameter pipe but not in a 2.54 cm (1 in.) diameter pipe.

### BAF Process Streams

The compositions of the process streams of the BAF process and their propagation results are listed in tables 6 and 7, respectively. The tests were conducted over a range of diameters from 2.54 to 15.875 cm (1 to 6 1/4 in.). In no instance did any of the streams propagate.

### Guanidine Nitrate - Guanidine Nitrate/Water

The detonation rates of technical grade guanidine nitrate are in table 8. Sustained high order detonations occurred during all six tests. The average rate of detonation was 2,762 m/sec. The effect of its dilution with water on the propagation characteristics of guanidine nitrate (table 9) indicates that propagation in 5.08 cm (2 in.) pipes did not occur when water constituted 25% or more of the mixture.

## Nitroguanidine/Water

The propagation characteristics of nitroguanidine/water mixtures are in table 10. The results obtained by diluting nitroguanidine with water are approximately the same as those obtained above for guanidine nitrate. In 5.08 cm pipes, propagation did not occur for water concentrations greater than 30%.

The process streams after dewatering and continuing through drying, for manufacturing both guanidine nitrate and nitroguanidine, contain less water than the critical levels required to sustain propagation. Accordingly, these streams deserve maximum attention with respect to safety design. Additional comments on design parameters are presented in the transition test paragraph and in reference 5.

## CONTRACTOR TEST STUDIES

Extensive sensitivity testing was conducted by the Hercules Powder Company under contract number DACA 45-71-C0121 for the Corps of Engineers (ref. 5) in accordance with CPIA publication No. 194 (ref. 4).

The testing (table 11) included impact, friction, electrostatic discharge, dust explosion, transition, and propagation tests on the reactor mixture charge, nitroguanidine, and guanidine nitrate, both pure and technical grade produced by the BAF process. The data indicates that these materials are relatively insensitive to the stimuli used on them.

### Impact Tests

All impact values were greater than the limits of the impact apparatus, except for the technical grade guanidine nitrate which contained 6 to 7% of ammonium nitrate and which accounts for its increased sensitivity.

### Transition Test (ref. 5)

A transition or critical height-to-explosion test is defined as the height of the material that will react explosively when initiated by flame. Experience has shown that as the diameter of the material increases, the corresponding critical height-to-explosion also increases. The transition test results show that, for all samples tested, the critical height-to-explosion for a 5.08 cm (2 in.) diameter is greater than 60.96 cm (24 in.). Since the reactors and precipitators have height/bed diameter ratio of less than 12, initiation of the materials in these areas of the process by impact, friction,

etc. would result in a fire and not an explosion. A ratio of more than 12 to 1 (height to diameter) would have to exist before an explosion could occur under these conditions.

#### Propagation Testing (ref. 5)

A propagation test determines the diameter of material that will propagate an explosive reaction when exposed to a shock stimulus. Table 11 shows that the critical diameter for the BAF reaction mixture, guanidine nitrate and nitroguanidine are >7.62 cm (3 in.) <2.54 cm (1 in.) and <1.27 cm ( $\frac{1}{2}$  in.), respectively. This means that the interconnecting pipelines after the reactors will not propagate an explosive reaction if the diameter is 7.62 cm (3 in.) or less, and that the guanidine nitrate and nitroguanidine dryers will propagate an explosive reaction if subjected to a strong shock source. However, there is no evidence that a shock source exists in the proposed system. As stated in the discussion of the transition tests, any ignition by impact, friction, etc. would result in burning and not an explosion. Therefore, a shock source would have to originate from outside the process, such as from high velocity projectiles, sabotage, etc.

Tables 12 and 13 present a summary of initiation, transition, and propagation test results on simulated compositions found in the mix tanks, dryers, and other operations associated with the U/AN process. The results were similar to those obtained from the BAF materials. These compositions are also relatively insensitive to impact, friction, and electrostatic discharge stimuli. The transition test results also indicate that a maximum height/bed ratio of 12 would result in a fire when subjected to stimuli such as impact, friction, and electrostatic discharge, propagation to explosion would require a strong shock from an external source.

#### CONCLUSIONS

1. For the U/AN process, the critical diameters determined by using shock initiation were 2.54 cm (1 in.) for the evaporator outlet and the liquid reactor outlet streams, and 3.81 cm ( $1\frac{1}{2}$  in.) for the mixed reactor feed stream.
2. The nitroguanidine conversion reactor outlet stream is not mass detonable.
3. Thermal analysis tests (DTA and TGA) on the process streams indicated in paragraph 1 above, showed no explosive behavior and produced controlled thermal decomposition only.



4. Propagation tests showed that the guanidine nitrate crystallizer stream in the U/AN process did not propagate; and, that the guanidine nitrate evaporator outlet U/AN process stream had a critical diameter of 2.54 cm.

5. Welland technical grade (fig. 10) guanidine nitrate yielded a detonation rate of 2,762 m/sec. Mixtures of guanidine nitrate and water did not propagate in 5.08 cm (2 in.) pipes when water was 25% greater in the mixture.

6. Mixtures of nitroguanidine and water did not propagate in 5.08 cm (2 in.) pipes when using a water concentration of 30% or greater.

7. Impact, friction, and electrostatic sensitivity show that the reactor mixture charge, nitroguanidine, and both pure and technical grade guanidine nitrate are relatively insensitive to these stimuli. Flame-initiated critical height tests indicate that these materials should not transcend to explosion within the process system. An external shock source, e.g., projectiles, sabotage, etc. would be required to stimulate an explosion.

## RECOMMENDATION

If the U/AN process is selected for future facilitation, additional propagation studies should be conducted, on the various process streams associated with it, to gain additional data for more definitive statistical inferences to establish more accurate design characteristics.

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Table 1. Critical diameter tests and results.

Comp. No.	Temp. °C	Pipe Assembly - Diam & Length	No of Tests	Results
1	135	1"Ø x 24", 3/4"Ø x 12" (2.54cm x 61cm, 1.91cm x 30.5cm)	12	DNP - No Sympathetic detonation
1	135	2"Ø x 10", 1½"Ø x 10", 1"Ø x 8" (5.08cm x 25.4cm, 3.8cm x 25.4cm, 2.5cm x 20.3cm)	2	Propagation stops at 1"Ø 2.54cm
CRITICAL DIAM OF COMP #1 is 1"				
2	130	2"Ø x 10", 1½"Ø x 10", 1"Ø x 8" (5.08cm x 25.4cm, 3.8cm x 25.4cm, 2.54cm x 20.3cm)	2	Propagation stops at 1½"Ø junction 3.81cm
2	130	1½"Ø x 20", 1"Ø x 12" (3.81cm x 50.8cm, 2.54cm x 30.5cm)	2	DNP
2	130	2"Ø x 20", 1½"Ø x 12" (5.08cm x 50.8cm, 3.81cm x 30.5cm)	2	Propagation stops at 1½"Ø junction 3.81cm
CRITICAL DIAM OF COMP #2 is 1½"				
3	190	2"Ø x 10", 1½" x 10", 1"Ø x 4" (5.08cm x 25.4cm, 3.81cm x 25.4cm, 2.54cm x 10.2cm)	1	No trace of pipe found
3	190	2"Ø x 10", 1½"Ø x 10", 1"Ø x 8", 3/4"Ø x 6" (5.08cm x 25.4cm, 3.81cm x 25.4cm, 2.54cm x 20.3cm, 1.91cm x 15.2cm)	1	No trace of pipe found
3	190	1"Ø x 10", 3/4"Ø x 10", ½"Ø x 8" (2.54cm x 25.4cm, 1.9cm x 25.4cm, 1.27cm x 20.3cm)	1	Propagation stops at 3/4"Ø junction 1.91cm
3	190	1"Ø x 10", 3/4"Ø x 10", ½"Ø x 8" (2.54cm x 25.4cm, 1.91cm x 25.4cm, 1.27cm x 20.8cm)	1	Propagation stops at 3/4"Ø junction
3	190	1"Ø x 20", 3/4"Ø x 12" (2.54cm x 50.8cm, 1.91cm x 30.5cm)	2	DNP through 1"Ø pipe 2.54cm
3	190	1½"Ø x 18", 1"Ø x 12" (3.8cm x 45.7cm, 2.54cm x 30.5cm)	2	Propagation stops at 1"Ø junction 2.54cm

Table 1. (continued)

Comp. No.	Temp. °C	Pipe Assembly - Diam & Length	No of Tests	Results
CRITICAL DIAM OF COMP #3 is 1"				
4	40	1½"Ø x 12"	1	Pipe blown apart-Probably due to booster
4	40	(3.81cm x 30.5cm) 2"Ø x 12"	1	Pipe blown apart-Probably due to booster
4	40	(5.08cm x 30.5cm) 2"Ø x 24"	2	DNP
4	40	(5.08cm x 61cm) 4"Ø x 12"	1	DNP - Pipe blown apart
4	40	(10.16cm x 30.5cm) 2"Ø x 24"	1	DNP - 8" pipe remains 20.3cm
4	40	(5.08cm x 61cm) 4"Ø x 36"	1	DNP - 10" pipe remains 25.4cm
H <sub>2</sub> O	40	(10.16cm x 91.4cm) 4"Ø x 26"	1	DNP - 10" pipe remains 25.4cm
4	40	(10.16cm x 68cm) 4"Ø x 36"	1	DNP - 14" pipe remains 35.6cm
4	40	(10.16cm x 91.4cm)		

Ø DENOTES DIAMETER - DNP - DOES NOT PROPAGATE

Table 2. (U/AN) GN/NQ process detonation study.

<u>PROCESS</u>	<u>STREAM</u>	<u>COMPOSITION</u>	<u>TEMP °C</u>	<u>MASS DETONABLE</u>	<u>CRITICAL DIAMETER IN.</u>
U/AN	EVAPORATOR OUTLET	13% GN 74% AN 13% U	130	YES	1 (2.54 cm)
U/AN	MIXED REACTOR FEED	9% GN 60% AN 31% U	120	YES	1½ (3.81 cm)
U/AN	LIQUID REACTOR OUTLET	33% GN 57% AN 10% U	180	YES	1 (2.54 cm)
U/AN AND BAF	NQ REACTOR OUTLET	56% H <sub>2</sub> SO <sub>4</sub> 7% WATER 21% NQ 2% GN 14% AS	40	NO	NA

Table 3. Hazard testing results.

Sample No.	Location in Pilot Plant	Temp. °C	Wt. %			Wt. % GN	Wt. %			PA App 2 Kg. wt., in.	Charge weight, gm	Friction Pendulum Steel Shoe
			AN	U	H <sub>2</sub> O		AN	U	H <sub>2</sub> O			
1	Centrifuge drier	158°F 70°C	1.0	0.0	5.0	94.0	1.0	0.0	5.0	40+	0.015	No reaction
2	Centrifuge	Ambient, 76°F	0.7	0.0	14.3	85.0	0.7	0.0	14.3	40+	0.017	No reaction
3	Quench Product	212°F 100°C	30.0	5.0	30.0	35.0	30.0	5.0	30.0	40+	0.033	No reaction
4	Evaporator Inlet	Ambient, 76°F	34.3	7.7	48.8	9.2	34.3	7.7	48.8	40+	0.026	No reaction
5	Evaporator Outlet	275°F 135°C	66.7	14.9	0.6	17.8	66.7	14.9	0.6	38	0.032	No reaction
6	Crystallizer Outlet	Ambient, 76°F	28.0	6.0	32.0	34.0	28.0	6.0	32.0			No reaction
			Sample No. A Shaken				Sample No. B Decanted			40+	0.031 (73°F)	
										40+	0.029 (74°F)	

Table 4. DTA tests on stream compositions.

Sample			Observed	Observed	Remarks
Contents(%)			Endotherms (°C)	Exotherms (°C)	
I.	Evap Feed Outlet	UREA (13%) NH <sub>4</sub> NO <sub>3</sub> (74%) Guanidine Nitrate (13%)	60°, 95° 120°, 135° 160°, 270° 300°	None	Sample vaporized away Smoothly beginning at 160°
II.	Mixed Reactor Feed	UREA (31%) NH <sub>4</sub> NO <sub>3</sub> (60%) Guanidine Nitrate (9%)	60°, 72°, 90° 95°, 130° 260°, 300°	None	Extensive vapor- ization begins at 160° and is complete at 315°C.
III.	Liquid Reactor Outlet	UREA (10%) NH <sub>4</sub> NO <sub>3</sub> (57%) Guanidine Nitrate (33%)	60° 110° 260° 300°	330°	Frothing observed at 280°C. No violent reac- tion observed.
IV.	NQ De- hydration Reactor Outlet	Sulfuric Acid (56%) Water (7%) Ammonium Sulfate(14%) Nitroguanidine (Class II lot NCW 3-16 (21%) Guanidine Nitrate (2%)	None	150° 175° 320°	Smoke and vapori- zation. No violent reaction after cooling residue was solid.

Table 5. U/AN process stream composition.

<u>Operation</u>	<u>GN Crystallizer</u>	<u>GN Evaporator Outlet</u>		
	<u>Diameter</u>	<u>Diameter</u>		
	<u>2 Inch</u>	<u>2 Inch</u>	<u>2 Inch</u>	<u>1 Inch</u>
<u>Ingredients</u> %				
Guanidine Nitrate (GN)	34.0	17.8	18.5	18.5
Ammonium Nitrate	28.0	66.7	66.6	66.6
Urea	6.0	14.9	14.9	14.9
Water	32.0	0.6	--	--
Temp.			250°F	250°F
	DNP*	Complete Detona- tion	Sustained Propaga- tion	DNP*

\*DNP - Did not propagate



Table 6. Composition of BAF process streams.

<u>Stream Identification</u>	<u>Stream Composition, Wt %</u>				
Operation					
Number	<u>4</u>	<u>14</u>	<u>94</u>	<u>100</u>	<u>116</u>
<u>Ingredients</u>					
Calcium Nitrate	18.6	25.8	0.25		0.40
Guanidine Nitrate	16.7	20.5	13.9	13.3	7.91
Ammonium Nitrate	46.2	39.0	46.53	44.6	74.96
Water	12.3	11.4	39.24	42.1	16.73

#4--R-202--Primary Reactor--Temp 250°F (121°C)

#14--R-205--Fourth Stage Reactor--Temp 250°F (121°C)

#94--L 236 A & B--Crystallizers--Temp 70°F (21°C)

#100--Stream--Temp 70°F (21°C)

#116--E-234--Evaporator--Temp 338°F (170°C)

Table 7. Propagation characteristics of BAF process streams.

<u>Stream Number</u>	<u>Nominal Diameter, Inches</u>					
	1	2	3	4	5	6½
4		N				
14	N	N	N			
94			N			
100		N				
116			N			
BAF Reactor Cold Melt				N	N	N

N - Did not propagate or incomplete propagation.

Table 8. Detonation velocity of technical grade guanidine nitrate - witness plate data.

Pipe Size 1" (2.54 cm)							
No.	Alum (62 mls)	Approx. Diam. of Depression inches	Guanidine Nitrate(GMS)	Approx. Depth of Depression inches	Time Over lft in Msec (1 x 10 <sup>-6</sup> )	Rate m/sec	Results
1	No	1.125 2.86 cm	140	.195 .50 cm	111.2	2,741	Hi Order Lg Frag
2	No	1.125 2.86 cm	146	.175 .44 cm	109.2	2,791	" "
3	No	1.063 2.70 cm	148	.165 .42 cm	108.6	2,807	" "
4	Yes	1.000 2.54 cm	145	.130 .33 cm	109.7	2,778	" "
5	Yes	0.750 1.91 cm	146	.100 .25 cm	111.6	2,731	" "
6	Yes	1.000 2.54 cm	142	.120 .30 cm	112.0	2,721	" "
						2,762 m/s Avg.	

Table 9. Propagation characteristics of guanidine nitrate/  
water mixtures.

<u>Mixture Composition, Wt %</u>		<u>Propagation Test Results</u> <u>Pipe, Diameter, in.</u>			
<u>GN</u>	<u>H<sub>2</sub>O</u>	<u>1.0</u>	<u>1½</u>	<u>2</u>	<u>2½</u>
100	0	Y	Y	Y	Y
95	5	N*			
85	15	N	Y*		
80	20			Y	
75	25			N	
70	30			N	
60	40			N	

\*N - Did not propagate or incomplete propagation

Y - Detonated completely

Table 10. Propagation characteristics of NQ/H<sub>2</sub>O mixtures.

Mixture Composition, Wt %		Propagation Test Results Pipe Diameter, In.				
<u>NQ</u>	<u>H<sub>2</sub>O</u>	<u>1.27</u> <u><math>\frac{1}{2}</math></u>	<u>3.81</u> <u><math>1\frac{1}{2}</math></u>	<u>5.08</u> <u>2</u>	<u>6.35</u> <u><math>2\frac{1}{2}</math></u>	<u>7.62</u> cm <u>3</u> in.
80	20	*Y				Y
70	30	N		N		Y
60	40			N		
40	60			N		

\*N - No Incomplete

Y - Yes

Table 11. Test results for NG plant (BAF process).

Sample	Temperature	Impact	Friction	ESD	Dust Explosion	Transition	Propagation
Ca(NO <sub>3</sub> ) <sub>2</sub> 26.6%	125°C	≥ 211,500 ft-lb/sec	≥ 65,625 psi @ 8 ft/sec	5.0 Joules ambient (ambient)	≥ 4.1 <sub>3</sub> oz/ft <sup>3</sup> (4.1 kg/m <sup>3</sup> )	> 24" @ 1" ID (61cm @ 2.54cm) > 24" @ 2" ID (61cm @ 5.1cm)	1" - NP (2.54cm) 2" - NP (5.1cm) 3" - NP (7.6cm)
NH <sub>4</sub> NO <sub>3</sub> 40.2%		(287 kJ/s)	(452 MPa @ 2.4 m/s)				
GN 21.2%							
H <sub>2</sub> O 12%							
CaCN <sub>2</sub>	Ambient						
NQ	Ambient	≥ 77.6 ft-lb/ in <sup>2</sup> (175 kJ/m <sup>2</sup> )	≥ 104,761 psi @ 8 ft/sec (722 MPa @ 2.4 m/s)	0.5 Joules	≥ 4.1 <sub>3</sub> oz/ft <sup>3</sup> (4.1 kg/m <sup>3</sup> )	> 24" @ 2" ID (61cm @ 5.1cm) (SCH 40 Con- finement)	CF < 1/2" (SCH 40 Confinement)
GN-Pure	Ambient	≥ 59.7 <sub>2</sub> ft-lb/ in <sup>2</sup> (135 kJ/m <sup>2</sup> )	≥ 122,400 psi @ 8 ft/sec (844 MPa @ 2.4 m/s)	1.26 Joules			
GN-Technical Grade	Ambient	31.6 <sub>2</sub> ft-lb/ in <sup>2</sup> (71 kJ/m <sup>2</sup> )	≥ 105,800 psi @ 8 ft/sec (729 MPa @ 2.4 m/s)	0.075 Joules	≥ 4.1 <sub>3</sub> oz/ft <sup>3</sup> (4.1 kg/m <sup>3</sup> )	> 24" @ 1" ID (61cm @ 2.54cm) > 24" @ 2" ID (61cm @ 5.1cm)	< 1" (1" = 2980 m/sec (2.54cm) (2 1/2" - 3780 m/sec) (6.4cm)
Dryer Configuration Propagation Tests 6" (15.2cm) Deep x 12" (30.5cm) Wide x 24" (61cm) Long							
GN							
1" booster - no propagation (2.54cm)							
2" booster - propagation 2900 m/sec (5.1cm)							

Table 12. Summary of initiation test results.

<u>Sample Mixture</u>		<u>Simulated Mix</u>	<u>Temperature</u>	<u>Impact<sup>2</sup></u> (ft-lbs/in <sup>2</sup> )	<u>Friction</u> (@ 8 fps)	<u>ESD*</u> (Joules)
AN	U	GN				
1	1	-	Mix Tank	Ambient	> 69,000 psi (475 MPa)	
2	1	-	Mix Tank	Ambient	≥ 69,000 psi (476 MPa)	
4	1	-	Mix Tank	Ambient	≥ 67,000 psi (462 MPa)	0.5
4	1	-	Mix Tank	135°C	39,090 psi (269 MPa)	
		Pure	Dryer	Ambient	≥ 122,400 psi (844 MPa)	1.26
		Technical Grade	Dryer	Ambient	> 105,800 psi (729 MPa)	0.075
				31.6 (71 KJ/m <sup>2</sup> )	≥ 122,800 psi <u>2/</u> (844 MPa)	
4.5	1.5	4.0	Eutectic Tank	60°C	45,614 psi (314.5 MPa)	

(1) Rate term since sample under test was in a liquid phase. (ft-lbs/sec)

(2) At 10 fps.

\* ESD - Electrostatic Discharge

Table 13. Transition and propagation results.

Sample	Initiation Source	Simulated Mix	Test Sensing	Temperature	Critical Ht.		Velocity (m/sec)	Remarks
					Container Size (2) (in.)	Diam. (in.)		
Technical Grade GN	12 gm bag igniter	Dryer	Visual	Ambient	TRANSITION 1 x 24 (2.54cm x 61cm)	$C_H \geq 24$ (61cm)	-	Smoke, muffle noise sample scattered
Technical Grade GN	"	"	"	"	2 x 24 (5.1cm x 61cm)	$\geq 24$ (61cm)	-	Smoke, muffle noise sample scattered
AN/U/Silica Gel 4/1/1	Pyrofuze (Al-Pd-alloy	Reactor tube after 2 hrs of heating	"	180°C	2 x 24 (5.1cm x 61cm)	$\geq 24$ (61cm)	-	No reaction, sample left in pipe
Technical Grade GN	Comp C-4 (1)	Dryer	Probe	Ambient	1 x 24 (2.54cm x 61cm) 2 1/2 x 24 (6.4cm x 61cm)	<1 (2.5cm)	2980 3780	Sample consumed
AN/U/Silica Gel 4/1/1	"	Reactor tube after 2 hrs of heating	"	180°C	1 x 24 (2.54cm x 61cm)	<1 (2.5cm)	-	Propagation
GN/AN 5.4/4.6	"	Reactor tube	"	180°C	1 x 24 (2.54cm x 61cm)	<1 (2.5cm)	-	Propagation
AN/U 4/1	"	Mix Tank	"	100°C	1 x 24 (2.54cm x 61cm)	1 (2.5cm)	1200-1300	Started, then stopped 13" to 17" pipe left (33cm) (43cm)
GN/AN/U 3.4/3.8/2.8	"	Eutectic Tank	"	60°C	1 x 24 (2.54cm x 61cm)	>1 (2.5cm)	-	No propagation 17" to 20" pipe left (43cm) (51cm)

(1) Comp C-4 size - Diameter equal to pipe diameter and length 3 x diam.

(2) Pipe - Schedule 40, closed bottom for both tests, top capped for transition and top plate for propagation.



# NITROGUANIDINE PROCESS REACTIONS UREA/AMMONIUM NITRATE TECHNOLOGY

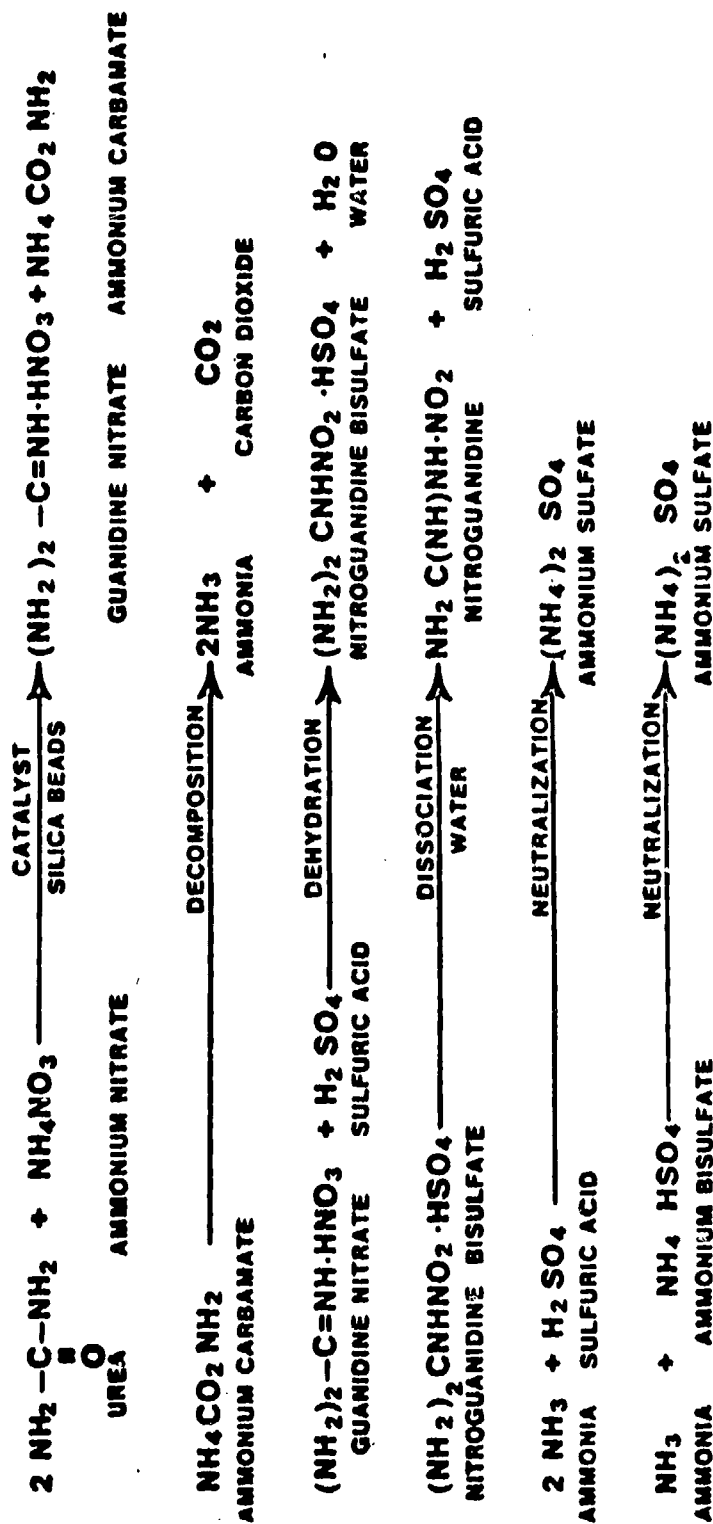


Figure 1. U/AN process reaction sheet.

# CHEMISTRY OF NITROGUANIDINE SYNTHESIS BAF PROCESS

## CALCIUM CYANAMIDE MANUFACTURE



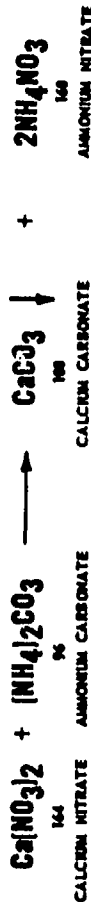
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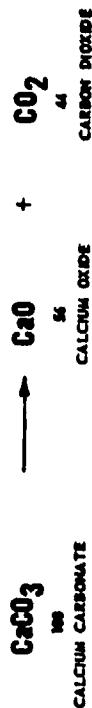
## AMMONIA RECOVERY



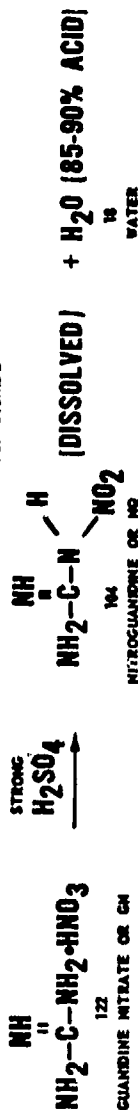
## CALCIUM REMOVAL



## CO<sub>2</sub> RECOVERY



## NQ MANUFACTURE



## NQ RECOVERY



NOTE: NUMBERS BELOW COMPOUNDS ARE MOLECULAR WEIGHTS

Figure 2. BAF process reaction sheet.





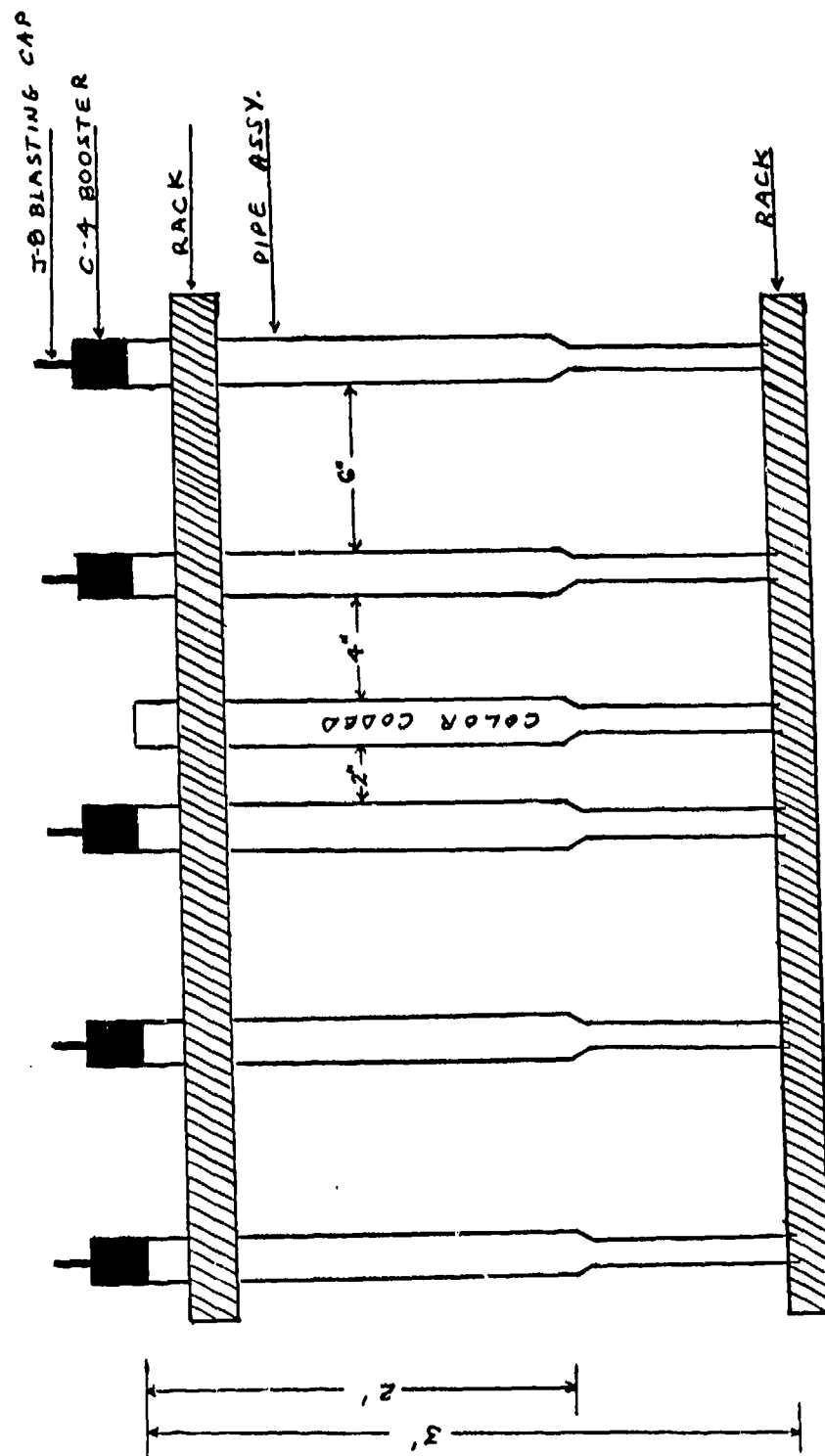


Figure 4. Critical diameter test apparatus.

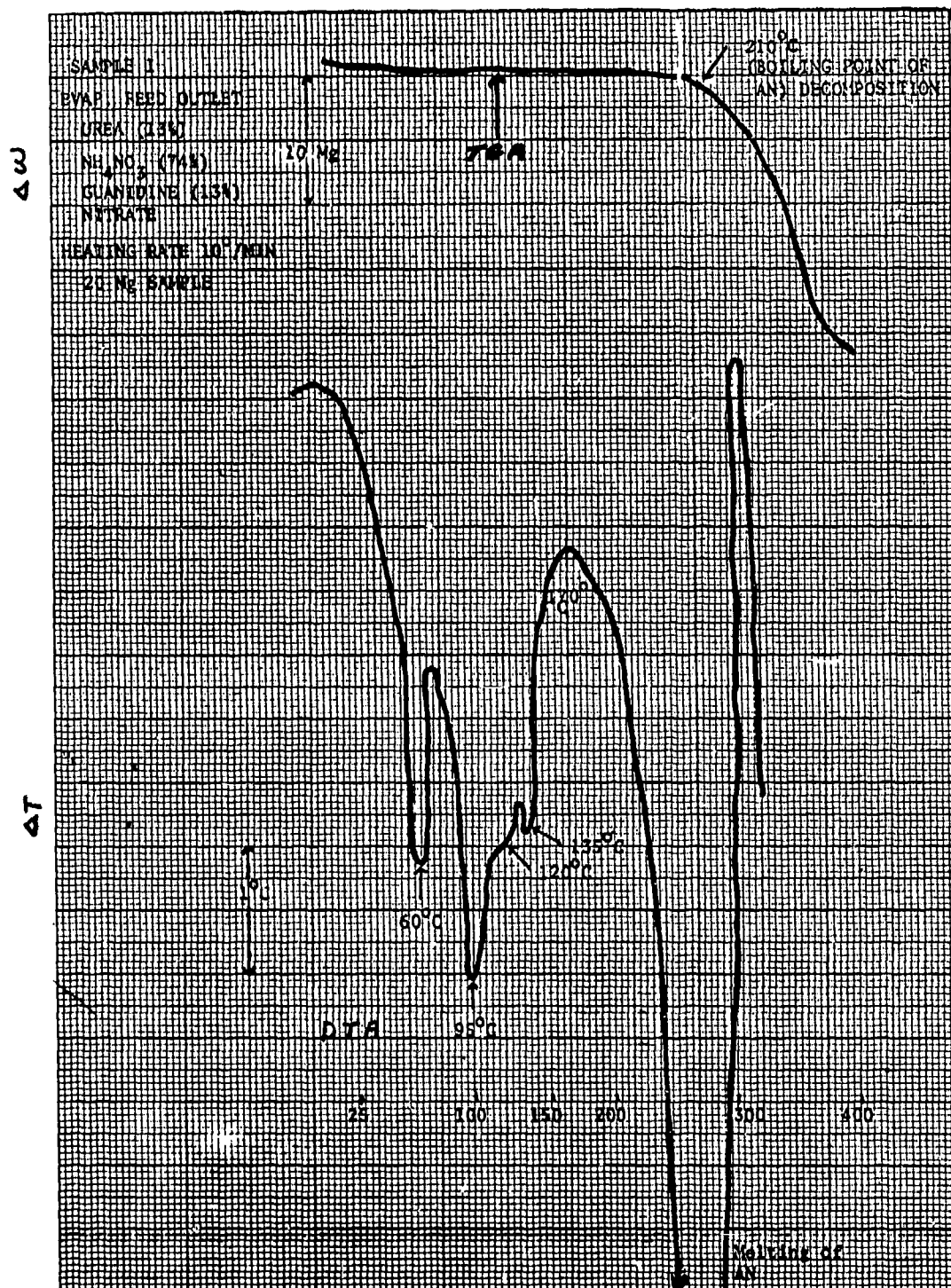


Figure 5. DTA and TGA - Sample I.

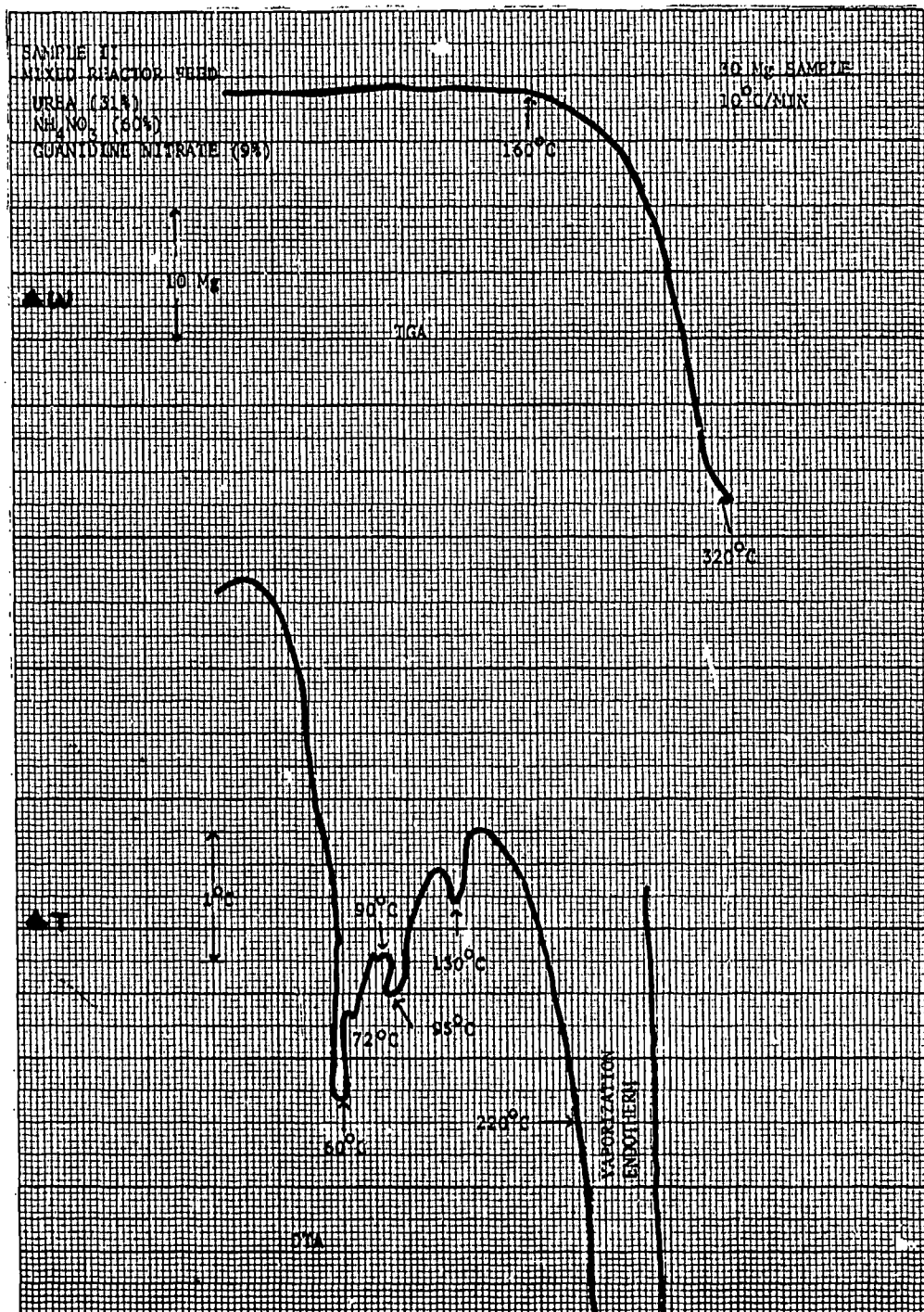


Figure 6. DTA and TGA - Sample II.

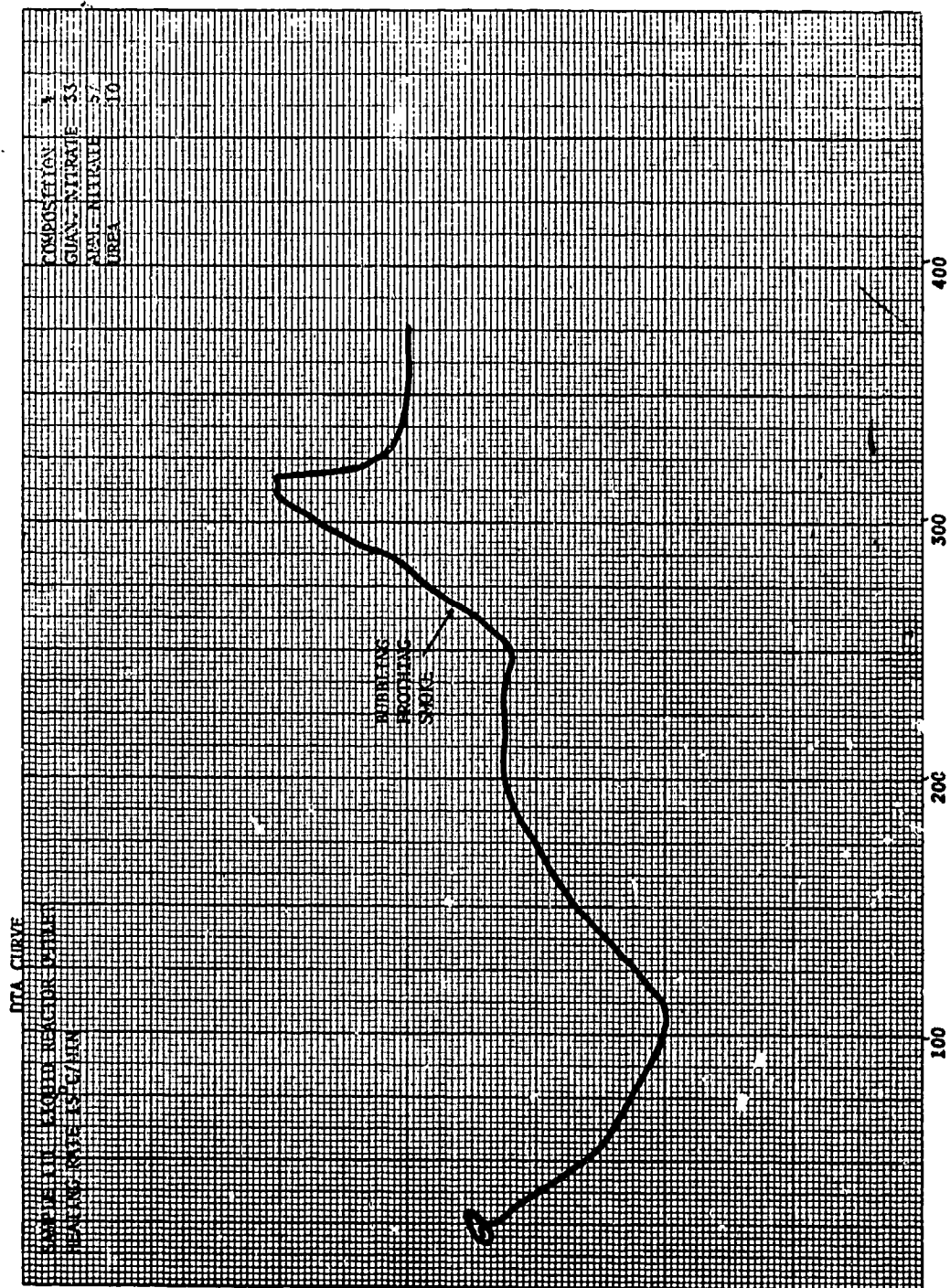


Figure 7. DTA - Sample III.



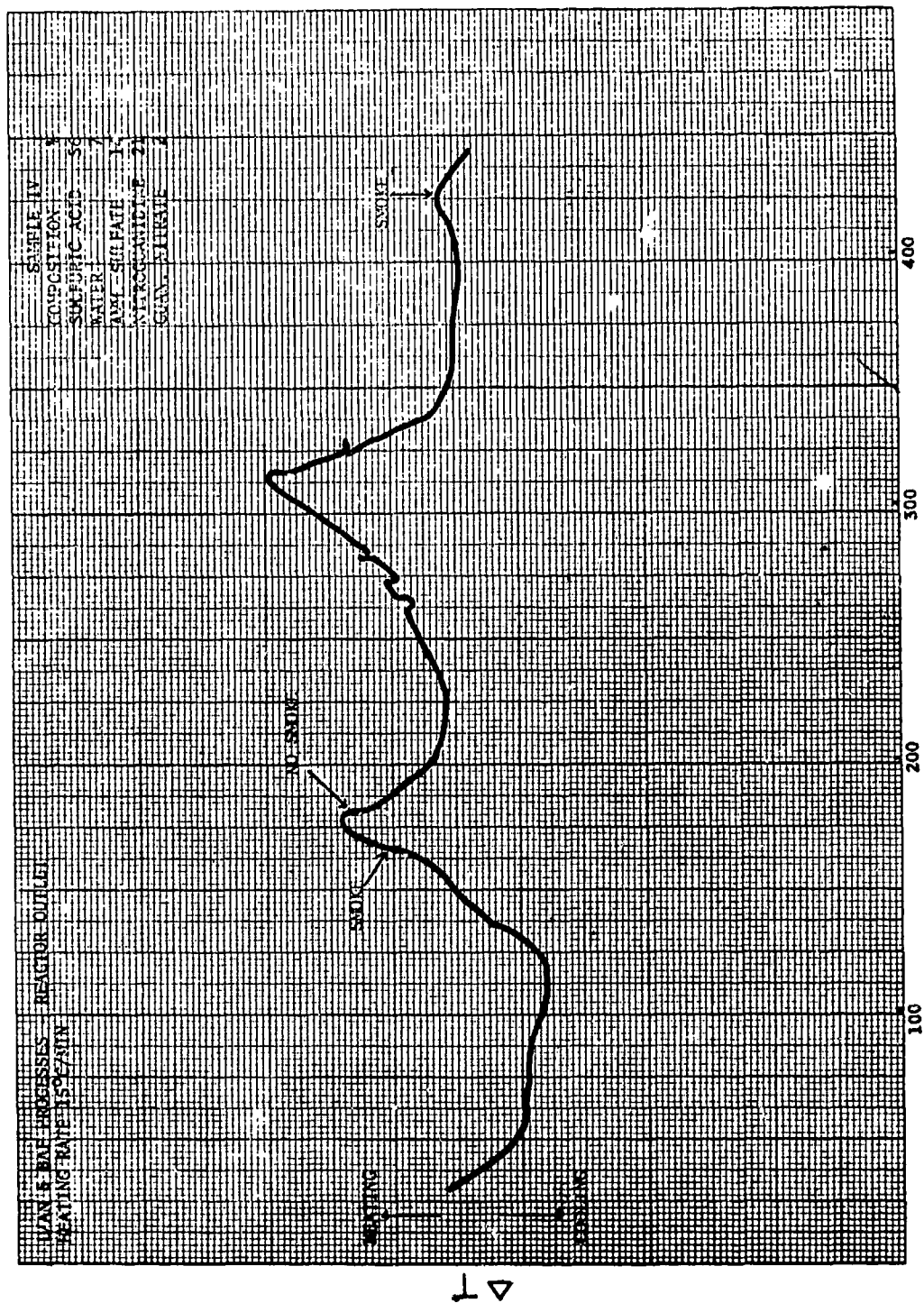


Figure 8. DTA - Sample IV.

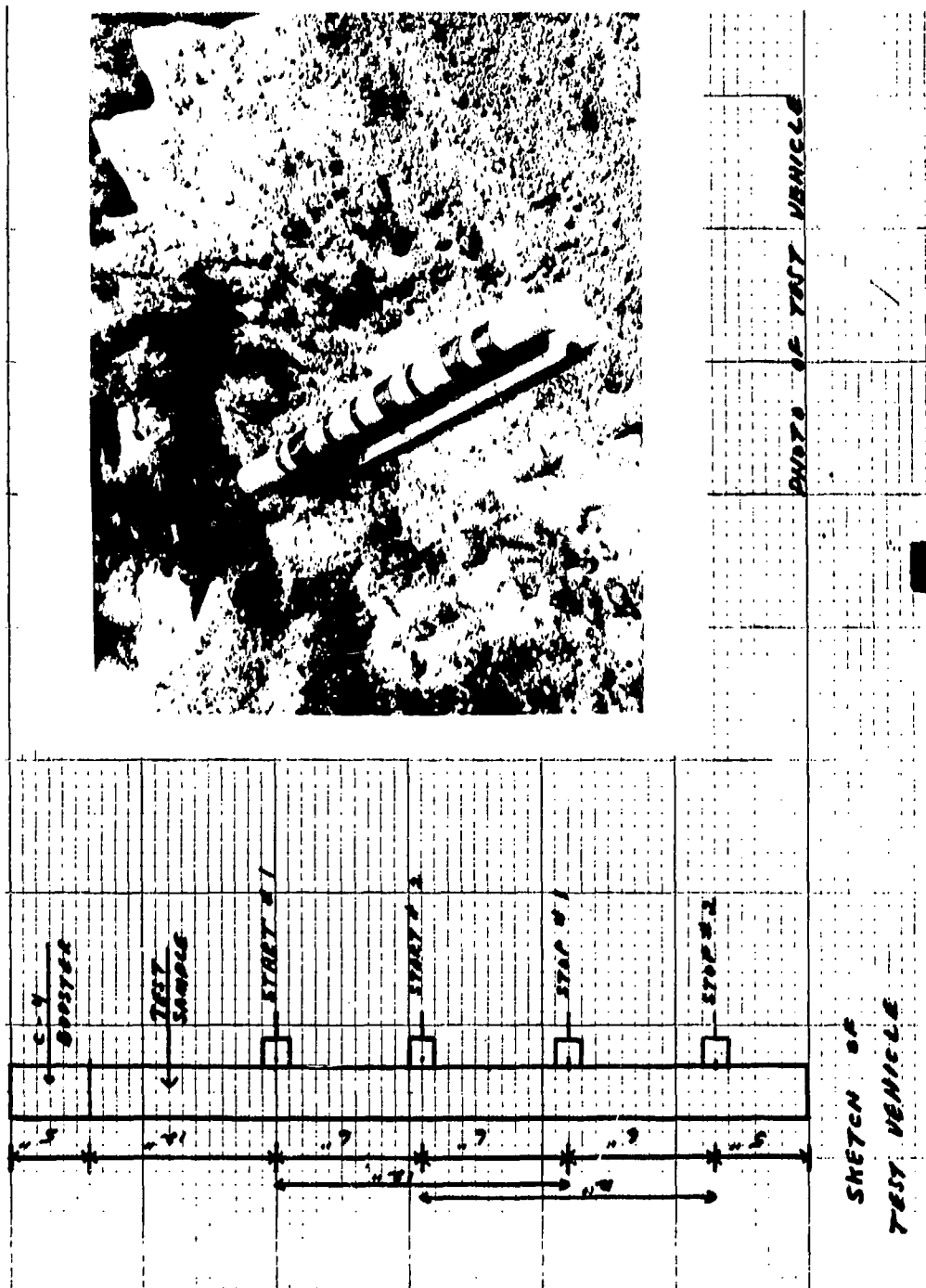


Figure 9. Propagation rate detonation apparatus.

# NITROGUANIDINE

## REACTIONS OF WELLAND PROCESS

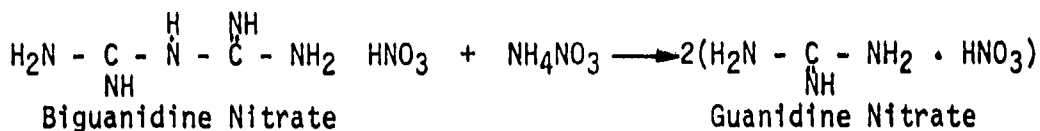
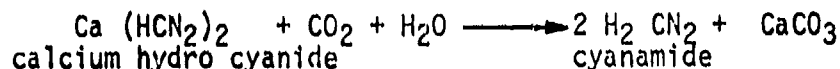
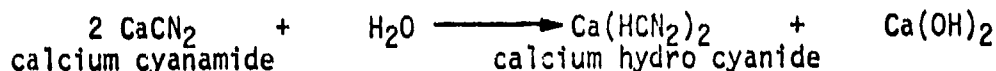
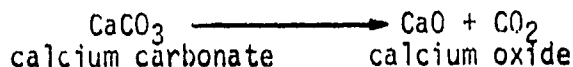


Figure 10. Welland process reaction sheet.

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